8. Sampling

Needs to Know Criteria
▪ Sampling requirements and frequencies
▪ Soil sampling and analysis
▪ Soil test reports
▪ Collection and analysis of plant tissue samples
▪ Role of plant tissue analysis in managing and monitoring cover crops at a land application facility
▪ Taking accurate samples and keeping good records
▪ Collection and analysis of ground water monitoring well samples
▪ Obtaining soil, wastewater, and crop tissue samples

Another important facet of proper system operation is sampling. Most wastewater land application permits require soil, plant tissue, effluent, and ground water sampling and analyses. If such monitoring is required, the system permit will specify which parameters to monitor, when and how often to monitor, and when results must be submitted. Although such sampling may not be required by every permit, all land application system operators should have a basic understanding of the different types of analyses available, proper sampling techniques, and how to interpret analysis results.

Sampling of the soil, plant tissue, effluent, and ground water are important to the proper operation of a land application site because the sample results tell the operator if the site is being managed properly and allows the operator to make informed decisions to avoid any possible adverse impacts to ground water, surface water, or public health.

8.1 Soil Sampling

It is not possible to look at a soil and predict if it is too acidic or if there are proper amounts of the essential nutrients present. Soils in Idaho vary in their need for nutrients, depending on soil characteristics, previous fertilization levels, and nutrient requirements of the crop.

The goal of soil testing is to learn enough about the soil to provide economically and environmentally sound nutrient recommendations and to help evaluate the operation and management of the land application site. Soil testing provides an approach for operators to assess soil pH and plant-available nutrients, to determine the need for chemicals and fertilizers, and to avoid losses and environmental damage from improper practices.

Refer to the University of Idaho Cooperative Extension System Bulletin 704, on Soil Sampling located in Appendix B for information about collecting soil samples. For information on how to analyze soil samples consult with the soil testing lab that will conduct the soil testing.

How Can a Soil Test Be Used to Determine the Land Limiting Nutrient?

In situations where wastewater application is limited by nutrients rather than by hydraulics, soil tests can be used to help determine the *land limiting nutrient*. The land limiting nutrient is the nutrient most likely to cause an adverse environmental or plant health effect if more is applied than the plants can use. Generally, nitrogen is found to be the land limiting nutrient, and application rates are based on supplying crop nitrogen (N) needs. The idea is to not apply N at rates greater than the crop can use, because the nitrate form of nitrogen can move through the soil and threaten ground water quality.

8.2 Plant Tissue Sampling

Healthy plants contain predictable concentrations of the essential elements. If these elements are not present in adequate amounts, then the plant suffers from a *nutrient deficiency*. In some cases, these nutrients are present in higher concentrations than required, and the plant may suffer from a *nutrient toxicity*. In either case, the plant is not healthy and is not efficiently removing nutrients from the soil. Plant tissue sampling can be used to distinguish between nutrient deficiency, nutrient sufficiency, and nutrient toxicity.

A recent soil test result can be helpful when interpreting a plant analysis. When visual symptoms of a suspected nutrient deficiency are present, take a soil sample at the same time from root zones of plants sampled. In this way, an evaluation of the soil in the affected area can be made along with an evaluation of the plant tissue. Sampling healthy and unhealthy plants, and their respective soil, is an effective crop management tool.

Plant analysis results can do the following:

- indicate the nutritional status of plants
- identify deficiencies and toxicities
- provide an accounting of nutrient utilization (crop uptake)
- provide a mechanism for optimizing yield, quality, and efficiency

Plant analysis assesses nutrient uptake while soil testing predicts nutrient availability. The two tests are complementary as crop management tools, but each has limitations. Soil testing is not always a good indicator of nutrients such as nitrogen and sulfur that leach easily.

A plant analysis may indicate that a nutrient deficiency or toxicity does not exist. Therefore, a factor other than nutrition may be responsible for poor plant growth or visual symptoms. In addition to sampling plant tissue for determining crop health, plant tissue sampling is also used to determine the amount of nutrients removed by the land application system. A sample of the harvested crop tissue is analyzed for nitrogen and phosphorus content to determine the amount removed from the field. Testing for ash may also be specified; ash results can be used to estimate the amount of total dissoved solids (TDS) removed from the field.

If plant tissue monitoring is required by your system permit, the permit will specify which parameters to monitor, when to monitor, and when results must be submitted.

Taking a Representative Sample

Proper sampling is the key to reliable plant analysis results. A sample can represent the status of one plant or 20 acres of plants. In general, a common-sense approach works well. Sampling instructions, information sheets, and shipping envelopes are provided at no charge and can be obtained at the county Cooperative Extension Service office.

When used for crop management, take samples from both "good" and "bad" areas. Comparison between the groups of samples helps pinpoint the limiting element. Comparative sampling also helps factor out the influence of drought stress, disease, or injury. Take matching soil samples from the root zones of both "good" and "bad" plants for the most complete evaluation.

When monitoring the status of healthy plants, take samples from a uniform area. If the entire field is uniform, one sample can represent a number of acres. If there are variations in soil type, topography, or crop history, take multiple samples so that each management area is represented by its own sample.

When monitoring for crop uptake of nutrients, take a sample that is representative of the harvested crop.

Selecting the Best Indicator Sample for Crop Management

The appropriate part of the plant to sample varies with crop, stage of growth, and purpose of sampling. For mature plants, the *most recent mature leaf* (MRML) is the best indicator sample, except as noted in Table 8-1.

The MRML is the first fully expanded leaf below the growing point. It is neither dull from age nor shiny green from immaturity. For some crops, the MRML is a compound leaf.

Table 8-1. Situations in which the most recent mature leaf (MRML) is not the best indicator sample.

Alfalfa, clover	Early growth through maturity – take top 2 to 3 inches of the plant.
Corn	Bloom through maturity – take ear leaf.
Small grain	Bloom through maturity – take the flag leaf.
Turfgrass	Take clippings from mower bag.

Choosing Sample Size

Laboratory analysis requires less than one gram of tissue, but, a good sample contains enough leaves to represent the area sampled. Therefore, the larger the area is, the larger the sample size needs to be.

Sample size also varies with crop. For crops with large leaves, a sample of three or four leaves is adequate. For crops with small leaves, a sample of 25 to 30 leaves is more appropriate. For most crops, 8 to 15 leaves is adequate.

Submitting the Sample

Do the following when submitting the sample:

- Use permanent ink or pencil on sample forms and envelopes. Tissue sample envelopes and information sheets are available from local Cooperative Extension offices.
- Pay attention to detail when filling out the information sheet. Supply the information requested. Note any conditions—drought, disease, injury, pesticide or foliar nutrient applications—that might be relevant.
- When identifying the plants that you sampled, give the exact name, if possible. Give each sample a unique identifier that will help you remember the plants or area to which it corresponds.
- When sending matching soil, solution, or waste samples, indicate the matching sample ID on the forms. Be sure the grower name and address are *exactly* the same on all matching information sheets.
- Ship all matching samples in the same container.
- Ship the tissue sample in a paper envelope or cardboard box, so it can begin drying during transport. Samples put in plastic bags will rot, and decomposition may alter test results.

Note: For additional discussion of plant tissue sampling, see Appendix C.

8.3 Wastewater Sampling

Wastewater analysis is the most accurate and efficient way to measure the levels of nutrients and other constituents in the land applied wastewater. Because the amounts of these constituents can vary among wastewater streams, laboratory analysis lets an operator know the proper amount of wastewater to apply to meet the specific plant and general management needs for each site.

When management decisions are made without wastewater analysis information, even well-intentioned users can reduce plant growth and yields or endanger the environment. Typically, monthly sampling and analysis of the land applied wastewater is required in the land application permit. The sample collection location and constituents to be analyzed are specified in the permit and vary between facilities, depending upon site wastewater characteristics and management practices.

Wastewater land application operators who fail to test wastewater are faced with a number of questions they simply cannot answer:

- Are they supplying plants with adequate nutrients?
- Are they building up excess nutrients that may ultimately move to streams or ground water?
- Are they changing the soil pH to levels that will not support plant production?

- Are they applying heavy metals at levels that may be toxic to plants and permanently alter soil productivity?

Because environmental damage and losses in plant yield and quality often happen before visible plant symptoms, wastewater land application operators should always have their wastewater analyzed by a competent laboratory, and they are encouraged to have their application rates evaluated by a knowledgeable agronomist.

Wastewater Sampling Terminology

A wastewater land application system operator should be familiar with the following wastewater sampling terms:

- *Grab Sample* - A grab sample is a sample collected over a period not exceeding 15 minutes. A grab sample is normally associated with water or wastewater sampling. However, soil, sediment, etc., may also be considered grab samples; no particular time limit would apply for the collection of such samples. Grab samples are used to characterize the medium at a particular instant in time and are always associated with instantaneous water or wastewater flow data, where appropriate. Grab sampling is conducted when the following is true:
    - the wastewater stream is not continuous
    - the characteristics of the wastewater stream are known to be constant
    - the sample is to be analyzed for parameters whose characteristics are likely to change significantly with time (e.g. dissolved gases, bacteria, etc.)
    - the sample is to be collected for analysis of a parameter where the compositing process could significantly affect the observed concentrations
    - data on maximum/minimum concentrations are desired for a continuous wastewater stream
    - when the permit monitoring requirements specify grab collections
- *Composite Sample* - Composite samples are used when average concentrations are of interest and are always associated with average flow data (where appropriate). Composite sampling is employed when the wastewater stream is continuous or it is necessary to calculate mass/unit time loadings or when analytical capabilities are limited. Many wastewater land application permits require composite sampling of the land applied wastewater.
- *Split Sample* - A split sample has been portioned into two or more containers from a single container. Portioning assumes adequate mixing to assure the "split samples" are, for all practical purposes, identical.
- *Duplicate Sample* - Duplicate samples are collected simultaneously from the same source, under identical conditions and into separate containers.

- *Control Sample* - A control sample is collected upstream or upgradient from a source or site to isolate the effects of the source or site on the particular medium being evaluated.
- *Background Sample* - A background sample is collected from an area, water body, or site similar to the one being studied but located in an area known or thought to be free from pollutants of concern.
- *Sample Aliquot* - A sample aliquot is a portion of a sample that is representative of the entire sample.

Sampling Procedures

Proper sampling is the key to reliable wastewater analysis results. Although laboratory procedures can be extremely accurate, they have little value if the samples fail to represent the wastewater stream. The importance of careful sampling becomes clear when one recognizes that laboratory determinations are often made on a portion of the sample submitted that is as little as 0.02 pound, (1 gram) for solid materials, or less than a tablespoon (10 milliliters) for liquid materials.

Important considerations for obtaining a representative wastewater sample include the following items:

- If possible, the sample should be collected where the wastewater is well-mixed. Therefore, the sample should be collected near the center of the flow channel, at a depth of approximately half the total depth, where the turbulence is at a maximum and the possibility of solids settling is minimized. Skimming the water surface or dragging the bottom should be avoided.
- In sampling from a mixing zone, cross-sectional sampling should be considered. Dye may be use as an aid in determining the most representative sampling points.
- If manual compositing is employed, the individual sample bottles must be thoroughly mixed before pouring the individual aliquots into the composite container.

8.4 Ground water Sampling

Ground water monitoring is a standard requirement of most wastewater land application permits to ensure that deterioration of ground water quality does not occur. One of the most critical parts of ground water monitoring is the sample collection process. The best lab in the state cannot produce accurate results from a sample that was contaminated before the lab received it. It would take a lot of time to cover all of the different types of monitoring systems and wells.

Note: For additional guidance on ground water well monitoring see the DEQ *Guidance for Land Application of Municipal and Industrial Wastewater,* on the DEQ Web site at http://www.deq.idaho.gov/water/permits_forms/permitting/guidance_wlap.pdf

The following discussion concentrates on the most common monitoring systems for land application of wastewater. However, no matter what type of well is being sampled, the procedures and important concepts covered should be followed consistently throughout the monitoring process.

Ground water is sampled from specially designed wells that are carefully located. The wells must be sampled immediately after construction and before land application begins—this determines background levels of constituents that are to be monitored. The system permit gives the frequency for all sampling based on facility and site conditions.

Before beginning compliance monitoring, you should do the following:

- determine the sampling schedule and parameters to be monitored as specified in your permit
- select a laboratory that can meet your permit requirements
- provide the lab with a copy of the monitoring requirements in your permit

8.5 Equipment and Supplies

Prior planning and careful preparation of field equipment before sampling will ensure good results from the laboratory. The following is a list of supplies and equipment to be used when sampling ground water:

- disposable gloves
- documentation (forms, log books, and O&M manual, etc.)
- indelible ink pen
- well lock keys
- tape measure
- water level monitoring device and supplies (batteries, chalk and paste as needed)
- field parameter meters with calibration standards
- decontaminated sampling pump with proper tubing and power supply
- bailers with line
- sample bottles
- sample labels
- packing tape
- stop watch
- graduated cylinder
- filtration equipment
- cooler with cold packs or ice
- cleaning buckets and containers

- plastic garbage bags
- small sealable plastic bags or containers
- plastic sheeting
- paper towels and hand soap
- cleaning brushes
- phosphate-free laboratory soap
- deionized organic-free water and hand sprayers
- high purity laboratory grade hexane, acetone, or isopropanol (all available from laboratory supply companies)

Customized kits for sample collection may be supplied by your laboratory. These kits include all the items needed for collection and shipment of samples. Follow laboratory instructions and read container labels. Be careful not to discard preservatives that may have already been added to some containers.

If you are not using a kit, use only new containers or sanitized reusable containers, supplied by a lab, of the appropriate types for the required parameters. Select and prepare them according to your laboratory's instructions. Label sample containers before sample collection and record the type and amount of preservative required on each sample label. Ensure that all sampling equipment, such as bailers, containers, and tubing has been selected and thoroughly cleaned based on the parameters to be monitored. Disposable bailers of the appropriate composition may be used. Use Teflon™, stainless steel, or glass when sampling for organics, such as solvents and petroleum product contamination. Do not use PVC or other plastics.

8.6 Minimizing Contamination Risks

Minimize contamination risks while collecting samples by doing the following:

- ensure that all sampling equipment (bailers, tubing, containers, etc.) has been thoroughly cleaned and selected based on compatibility with parameters to be monitored
- use Teflon, stainless steel, or glass when sampling for organics; do not use PVC or other plastics
- use Teflon or glass when sampling for trace metals
- use new sample containers when sampling for compliance monitoring; do not reuse containers
- keep containers closed before filling, and do not touch the inside of containers or caps
- wear a new pair of disposable gloves or decontaminated reusable gloves for each sampling site
- place new plastic sheeting on the ground near each well to hold the sampling equipment; do not step on the sheeting

- place small samples that require cooling, such as volatile organics, in sealable plastic containers immediately after collection and before submerging in ice
- do not smoke while collecting or handling samples, because volatile residues in the smoke can cause sample contamination
- do not leave your vehicle running near the sample collection area, to prevent contamination from engine exhaust fumes
- when using a pump, set up the generator about 15 feet away and downwind from the well; perform all generator maintenance and fueling off-site and away from samples
- avoid unnecessary handling of samples
- if dedicated monitoring systems (those permanently installed in wells) are **not** used, clean equipment to be reused thoroughly before sampling each well to minimize the risk of cross contamination; bailers left in wells are **not** dedicated systems
- take enough pre-cleaned equipment to the field to sample each well, so that cleaning between wells is unnecessary; if field cleaning is necessary, an equipment blank may be used to make sure that no contamination results

Blanks should be used to check for contamination. Blanks consist of organic-free deionized water, which must be obtained from laboratories. Types of blanks include the following:

- a *trip blank* (a sealed container of organic-free, deionized water that must be taken to the field and sent back to the lab, unopened, with the samples); include at least one trip blank per cooler for volatiles to check for sample contamination during transportation
- a *field blank* consists of organic-free deionized water taken to the field and handled in the same manner as the samples to check for contamination from handling, from added preservatives, or from airborne contaminants at the site, which are not from the waste being disposed of at the treatment facility
- an *equipment blank* (organic-free deionized water, which is passed through the cleaned sampling equipment with added preservatives) may be used to detect any contamination from equipment used for more than one well

8.7 Measuring Static Water Level and Calculating Well Volume

To measure static water level, do the following:

1. From a permanent reference at the top of the well casing, lower a clean weighted steel tape or electric sounder into the well.
2. Record the wet level mark on the tape and subtract it from the reference point to obtain the depth of water. (Use the same reference point each time a water level measurement is made at the well.)

To calculate the well volume, do the following:

1. Subtract the depth to water level measurement from the known total depth of the well to obtain the height of the water column.
2. Calculate well volume in gallons by multiplying the inside area of the well (measured in square feet) times the height of the water column (measured in feet) times 7.48 gallons per cubic foot. Or use Table 8-2 for a quick conversion of well volume in gallons.

Table 8-2. Well Diameter Conversion Table.

Well Casing Diameter	Gallons per foot of Water Column Height
2 inches	0.163
4 inches	0.652
6 inches	1.5
8 inches	2.6

8.8 Purging the Well

Before collecting any ground water samples, you must adequately remove stagnant water from the well by doing the following:

1. To purge a sufficient amount of water from a well, you must first calculate the well volume.
2. Based on the calculated well volume, pump or bail at least three well volumes from the well and/or until measurements for pH, specific conductance, and temperature meet the following conditions:
    - two successive temperature values measured at least five minutes apart are within one degree Celsius of each other
    - pH values for two successive measurements, measured at least five minutes apart, are within 0.2 units of each other
    - two successive specific conductance values, measured at least five minutes apart, are within 10% of each other.

This procedure will determine when the well is suitable for sampling for constituents required by the permit. Other procedures, such as low flow sampling, may be considered by DEQ for approval.

Following well recovery, samples may be collected using low rate pumping devices or a bailer. Dispose of purged water appropriately, according to state and federal regulations.

Purging with a Pump

Low rate pumping is the preferred method for purging, because bailing may increase turbidity by stirring up sediment in the well. When purging with a pump, do the following:

1. Slowly lower the pump to just below the top of the standing water column.

2. Continue lowering it as the water level drops and the stagnant water is removed, as explained in the previous section .

Purging with a Bailer

Using low flow pumps for purging generally produces high quality representative samples. However, if a pump is not available or cannot be used, use a bottom-emptying bailer to purge and collect samples. Bailer lines of braided nylon or cotton cord must not be reused, even if clean, to avoid the probability of cross-contamination. Lines must consist of Teflon-coated wire, single strand stainless steel wire, or other monofilament line.

Note: Do not leave bailers in wells. Contamination can occur when they are handled outside the wells and placed back inside. Contamination can also occur as a result of deterioration of bailer lines.

To purge using a bailer, do the following:

1. Lower the bailer slowly, to just below the water level, and retract slowly to reduce aeration and turbidity.
2. Collect the purged water in a graduated bucket to measure a minimum of at least three well volumes, or as discussed in the section above.

8.9 Collecting Samples

After wells are purged, collect samples using a low rate pump or a bottom emptying bailer. Sample containers obtained from commercial labs or laboratory suppliers may already contain the appropriate preservatives. Check with your laboratory and follow their instructions.

Sampling with a Portable Pump

When sampling with a portable pump, do the following:

1. Lower the pump, slowly, to the desired depth in the well. (Have sample containers ready before turning on the pump.)
2. Adjust the flow rate to less than 100 mL per minute to reduce agitation.
3. Decontaminate the pump before moving to the next well.

Sampling with a Bailer

To collect a sample with a bailer, do the following:

1. Lower the bailer slowly into the well, avoiding agitation, and allow it to fill.
2. Retract the bailer slowly, and discharge the sample carefully into the container until the correct volume has been collected.
3. Add preservative if required, cap the container, and mix according to laboratory instructions. Take precautions to minimize turbidity and sediment in samples.

4. Use purging and sampling techniques previously described to minimize turbidity and agitation of sediment in wells.

In low-yielding wells and those containing high levels of suspended solids, slowly lower a bailer to the lowest standing water level and allow the water to flow into it. Carefully lift the bailer out of the well without allowing it to scrape or bang against the well casing.

8.10 Minimum Cleaning Techniques

Portable sampling systems are used more frequently than dedicated systems because of lower costs. However, because portable systems require using the same equipment from well to well, they increase the possibility of cross contamination unless strict cleaning procedures are followed. Cleaning procedures must be selected based on the equipment composition and the parameters to be monitored.

The following is a summary of minimum cleaning techniques for bailers, applicable for other equipment of the same composition. For stainless steel bailers and equipment, use the following:

- phosphate-free soap and hot tap water wash
- hot tap water rinse
- deionized water rinse
- isopropyl alcohol rinse
- deionized water rinse
- air dry

Wrap the bailer with aluminum foil or other material to prevent contamination before use. Consider target contaminants when selecting a wrap material.

To clean Teflon or glass bailers and equipment use the following:

- phosphate-free soap and hot tap water wash
- hot tap water rinse
- ten percent nitric acid rinse
- deionized water rinse
- isopropyl alcohol rinse
- deionized water rinse
- air dry

Wrap to prevent contamination before use. Again, consider the target contaminants when selecting wrapping material.

8.11 Special Handling Procedures

Certain types of sampling require special handling procedures:

- For *trace metal analysis*, use extra care in selecting and cleaning all sampling equipment, including pumps, bailers, sample containers, etc.
- For *collecting volatiles*, use stainless steel or Teflon bailers , if using a pump for sample collection that has a flow rate that cannot be adjusted to less than 100 mL per minute, and do the following:
    1. Collect duplicate samples for volatile organics in special 40 mL septum vials, with Teflon lined disks in the caps to prevent contamination.
    2. Fill the vials to capacity, with no headspace, to prevent volatilization.
    3. Carefully pour the sample down the inside of the vials to minimize aeration and agitation until the containers are overflowing.
    4. Ensure that no air bubbles are trapped in the vials by applying the caps so that some overflow is lost.
    5. If bubbles are noted when the vials are inverted and tapped, set those aside to be discarded.
    6. Repeat the collection procedure using new vials. Include a trip blank of organics-free water, which must be obtained from your laboratory with each cooler containing samples collected for volatile organics.
- For *coliform samples*, collect directly into sterilized glass or sterilized plastic bottles that have been kept closed until ready to be filled. The sterilized containers often contain a preservative. Do not rinse prior to filling. Hold the bottles near the base until filled. Recap the bottles immediately, using care not to contaminate the bottles or lids. Store as required.

8.12 Filtering Samples

Adhere to the following requirements when filtering samples:

- Do not filter samples for coliforms.
- If there is a special request for the collection of a filtered sample for any analysis, use the appropriate type of filtering apparatus and filters.
- In low yielding wells and those containing high levels of suspended solids, lowering a bailer to below the static water level and allowing the well to recover into the bailer should produce a cleaner sample.
- Use purging and sampling techniques previously described to minimize agitation of sediment in affected wells and reduce the need for filtering. A pump used for purging and sampling will produce better samples from such wells.

8.13 General Procedures for Packing Ground Water Samples

Do the following when packing samples prior to shipment by courier or by personal transport to the laboratory:

1. Line a clean cooler with a large heavy duty plastic bag, and add bags of ice.

2. Place the properly tagged samples in individual, sealable plastic containers, and seal the containers with *chain-of-custody tape* to ensure sample integrity.
3. Place sample containers in the cooler, arranging bags of ice between samples to help prevent breakage; add sufficient ice to maintain the temperature of at 4^{o} C (39.2^{o} F) while the samples are in transit.
4. Enclose the appropriate forms in a sealable plastic container, place with samples in the chest, and seal the large bag with chain of custody tape.
5. Minimize transport time, and ensure that samples will reach the laboratory without being exposed to temperature variations and without exceeding holding times.

Once the laboratory has completed the sample analysis, a report containing the analytical results will be sent to the person requesting the analysis. Carefully fill out monitoring forms, making sure that all information is included and that the data transferred from laboratory reports are recorded in the correct concentration units. Include complete identification information, such as permit number and facility, or permit name, on all correspondence and additional laboratory reports. Be sure to submit the forms and lab reports on time.

You are the critical link in the ground water sampling process. It is vitally important that the procedures demonstrated be followed carefully to avoid costly resampling and to ensure that any ground water contamination is quickly detected and remediated.

Note: Failure to carry out any or all of these activities or comply with the terms and conditions of the permit is a violation of Idaho law and may subject the permittee to enforcement action and/or a civil penalty assessment.

If your facility uses a contractor for ground water sampling, you should still be familiar with the sampling frequencies and parameters and the general requirements of the sampling protocol. If you have any questions regarding your monitoring requirements, contact DEQ personnel in the appropriate regional office for your area.

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